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PETROGRAPHIC AND CHEMICAL PROPERTIES OF A LIGNITE FROM ESTEVAN, SASKATCHEWAN

(Report, 3 plates and 7 figures)

A. R. Cameron and T. F. Birmingham

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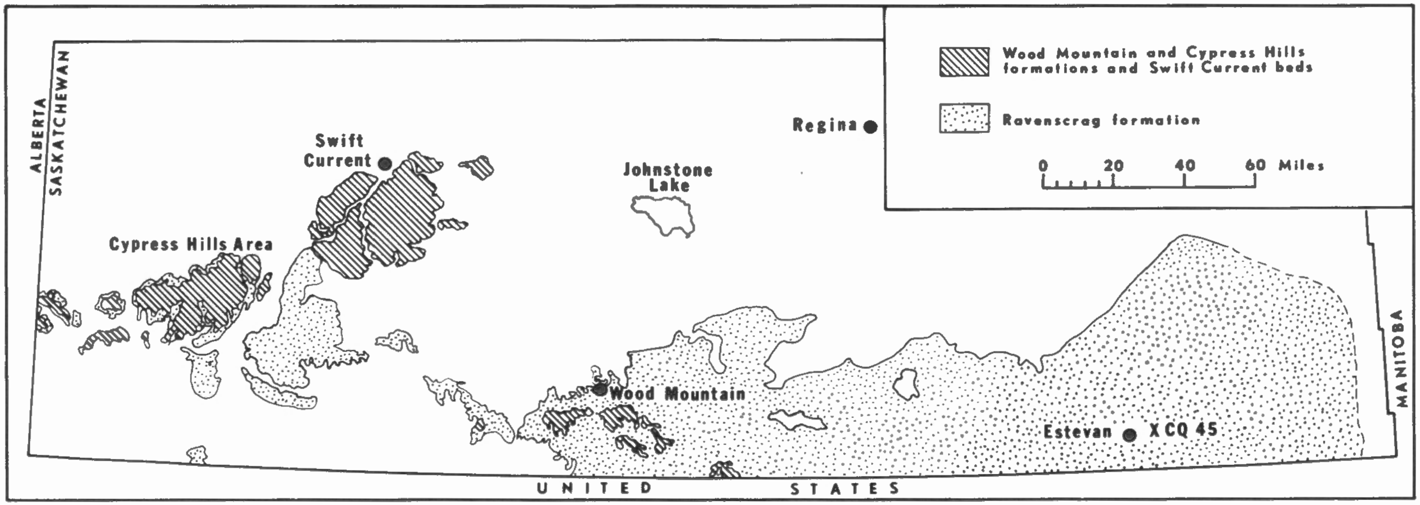
ABSTRACT

A column sample of a 98-inch-thick lignite seam from a strip mine near Estevan, Saskatchewan was analyzed petrographically and chemically. The maceral terminology applied was essentially that of bituminous coals except that three varieties of vitrinite were identified, namely, structured, structureless and groundmass. On the basis of petrographic characteristics, the seam was divided into ten intervals. Maceral variations among intervals showed a fairly high concentration of inertinite, especially fusinite and semifusinite, in the lower part of the seam. Chemical data showed sulphur contents to be well below 1 per cent in all intervals while ash contents varied from 6.8 to 8.6 per cent (dry basis) for the lower 88 inches of the seam. Volatile matter contents increased with an increase in the proportions of groundmass vitrinite, resinite and exinite. The petrographic data suggest a forest-moor origin for the seam with reed-moor and open-water influences gradually becoming stronger as seam accumulation progressed. Determinations of uranium, vanadium and molybdenum by intervals showed low concentrations. The highest uranium value was just under 3 ppm (total coal basis) obtained on the lowest interval of the seam. The best vanadium value was about 10 ppm (total coal basis) obtained on a high ash interval near the top of the seam. Molybdenum concentrations were all below 100 ppm on the ashed coal basis.



Frontispiece. Distribution of Ravenscrag Formation in Saskatchewan (adapted from MacKay, 1947).

PETROGRAPHIC AND CHEMICAL PROPERTIES OF A LIGNITE FROM ESTEVAN, SASKATCHEWAN

INTRODUCTION

An important part of the functions carried out by the Coal Research Section of the Geological Survey is the petrographic examination of the coals of Canada. Up to the present much of this work has been concerned with the bituminous coals, especially from Nova Scotia, Alberta and British Columbia. However the lower rank coals, such as the lignites of Saskatchewan, constitute an important unit, scientifically and commercially, in the coal series. The present report represents a first effort on the part of the Coal Research Section at characterizing a lignite petrographically. It is related in part to a study carried out on uranium content in western Canadian coals (Cameron and Birmingham, 1970) because of the possibility of a relationship between petrographic composition and uranium content.

During the course of the field investigations for radioactivity, a column sample of lignite was collected in the Klimax Mine of the Battle River Coal Company near Estevan, Saskatchewan. The sample was identified as CQ 45 and its location is shown in Figure 1. The seam at the point of sampling is 98 inches thick and is a well-compacted, blocky coal on the fresh face. Chemical analysis show it to be a lignite by ASTM standards.

The coal is Paleocene in age and occurs in the Ravenscrag Formation which extends more or less continuously across the southern part of Saskatchewan (see Fig. 1). It is considered to be equivalent in age to the Fort Union beds in North and South Dakota. Patches of younger rocks (Cypress Hills and Wood Mountain Formations and Swift Current beds) are found overlying the Ravenscrag beds, especially in western Saskatchewan.

According to MacKay (1947) the coal-bearing measures of the Ravenscrag have their maximum development at Estevan where eight seams occur in a stratigraphic interval of 750 feet. Farther west in the Wood Mountain area, and even more in the Cypress Hills area, there are fewer seams and they are equivalent apparently to the older seams in the Estevan section. The four upper seams in the Estevan area total 27 feet of coal and occur in about 125 feet of stratigraphic section.

Structurally the beds at Estevan are essentially flat lying when considered on a local basis. On a regional basis the Tertiary in southern Saskatchewan has been deformed by several gentle flexures as pointed out by MacKay (1947).

The authors acknowledge with thanks assistance received from the following individuals: W. J. Montgomery of the Mines Branch who arranged for the proximate and ultimate analyses, J. J. Lynch and K. A. Church of the Geological Survey of Canada for uranium, vanadium and molybdenum determinations and M. S. Barss also of the Geological Survey for photography.

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METHODS

The column collected in the field consisted of a series of blocks oriented with respect to top and bottom and labelled as to position in the seam. Each block was carefully wrapped in several plastic bags in the field to prevent excessive loss of moisture. In the laboratory a smaller complete column of the seam was cut from these blocks, mounted in an epoxy-type resin and impregnated with a mixture of Ambroid resin and acetone where such was deemed necessary to prevent the blocks from deteriorating. The blocks were then polished.

The petrographic analysis was done in two ways by microlithotypes and by macerals*. First the banded character of the coal was described in terms of microlithotypes. For this the polished blocks were used and the data plotted in the form of a log covering the entire thickness of the seam. By examination of this log it was then possible to divide the seam into distinctive petrographic intervals. From these intervals material was cut and crushed to -20 mesh and made into grain mounts, two for each interval. These were polished and studied for maceral content. Oil immersion was used at a magnification of 250 and 500 points were counted per pellet or 1,000 points per interval. In addition material from each interval was submitted to the Fuels Research Centre of the Mines Branch for proximate and ultimate analyses. Finally uranium, vanadium and molybdenum determinations were carried out on the ash of each interval.

TERMINOLOGY

The terms used in the petrographic description of high rank coals have been fairly well standardized. The same cannot be said for lower rank coals such as lignite. In fact petrographic classification for lignites is presently under study by a special committee of the International Committee for Coal Petrology.

The problems of petrographic classification are linked to the maturity of the coal, that is its rank. Much important work in this regard, especially with lignite, has been done in Germany. The Germans recognize three varieties of such coals, namely soft brown coal ("Weichbraunkohle"), dull hard brown coal ("Mattbraunkohle") and bright hard brown coal. ("Glanzbraunkohle") cited in ascending order of rank (Teichmüller and Teichmüller, 1967). Dull hard brown coal is described as being fairly solid, well bedded and dark in colour. Calorific values vary between 7200 and 9900 Btu/lb on the ash-free basis. The Estevan lignite by these criteria would fall into the category of dull hard brown coal.

Jacob (1961) discussed the petrographic description of soft brown coal and peat and the relation of such description to hard brown coal and bituminous coal. In bituminous coal the macerals can be placed in three main groups, namely vitrinite, inertinite and exinite. As a point of departure

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*Macerals may be considered the basic petrographic entities in coal. They are analagous to the minerals of inorganic rocks. Microlithotypes are assemblages of macerals determined microscopically. They are "rock types" in coal. Thus vitrinite and exinite are essential macerals of the microlithotype clarite just as quartz and feldspar are essential minerals of the rock type granite.

in his discussion Jacob used these three basic groups to relate those common aspects of petrographic composition which embrace a variety of coal ranks. For soft brown coal and peat he retained the terms inertinite and exinite although with considerable modification for the individual macerals within these groups, especially exinite. On the other hand he felt he could not apply the term vitrinite to soft brown coal. Instead he concluded from his observations that three different groups, which he designated as xylinite, detrinite and dopplerinite, probably represent the counterpart of vitrinite in the higher rank coals. He suggested that with increase in rank these materials become less diverse in their optical and chemical properties and eventually form the single vitrinite group. Xylinite includes those humic constituents whose most important characteristic is well preserved cell structure. Detrinite also includes humic materials in which the cell structure, though discernible in some varieties, is generally not prominent. Detrinite often occurs as a ground mass in which are found spore and pollen remains, resins, and particles of inertinite and on the whole it has a detrital or comminuted character. Dopplerinite shows little or no cell structure. It is apparently material which has undergone gelification and has a more homogeneous appearance than either xylinite or detrinite. Within each of these three groups Jacob described a number of macerals.

In discussing the hard brown coals Jacob emphasized their intermediate position between soft brown coal and bituminous coal. He felt that new terms were not necessary other than those already existing for bituminous coals and those which he had proposed for soft brown coal. He suggested that the terminology applied to hard brown coal should depend on the microscopic appearance of the coal, that is whether it appeared more like a bituminous coal or a soft brown coal. With the Estevan column these factors were taken into consideration in determining the maceral terms to be used. Microscopically the Estevan coal has many of the attributes of a bituminous coal although it is apparent that some of the features described by Jacob for soft brown coal are still evident, especially in regard to the "vitrinitic" constituent. For this constituent therefore, the ideas of Jacob suggested a three-fold breakdown, namely:

1. material with well defined structure,
2. material with little or no cell structure, a smooth texture, higher reflectivity and sometimes with cracks similar to those associated with dopplerinite,
3. material of lower reflectivity forming the groundmass for grains and particles of other macerals.

Because in general these components resemble more the constituents of bituminous coal than they do the xylinite, detrinite and dopplerinite of Jacob they have been designated as structured, structureless and groundmass vitrinite.

In the inertinite group fusinite, semifusinite and micrinite were identified. In the exinite group spore and pollen remains and cutinite were identified while under resinite was included cell-fillings and isolated bodies with the optical characteristics of both resins and waxes. Examples of the macerals identified are shown in Plates I through III.

For the microlithotype analysis the terms vitrite, clarite, duroclarite, clarodurite, carbargilite and fusite were used as defined in the Handbook (International Committee for Coal Petrology, 1963). It is of some interest that no durite was found.

RESULTS AND DISCUSSION

The results of the petrographic analyses are given in Figure 2. This figure shows on the left a seam profile in terms of microlithotypes summarized from the log. On the right of Figure 2 is shown the maceral data for the individual intervals. The seam profile shows rather distinctive petrographic intervals and this is also reflected in the plot of the maceralic data. In discussing petrographic descriptions the microlithotypes vitrite and clarite are considered "bright" components while duroclarite is considered intermediate and clarodurite, durite, carbargilite and fusite fall in the category of "dull" entities (Hacquebard _et al._, 1968). Strictly speaking, such descriptions refer to bituminous coals but because they imply a difference in composition as well as in lustre and because of the compositional continuity referred to earlier between ranks of coal they are applied in the present paper in connection with lignite.

The content of "bright" microlithotypes (vitrite plus clarite) varies in the present coal from 0-76 per cent. The latter percentage is in interval

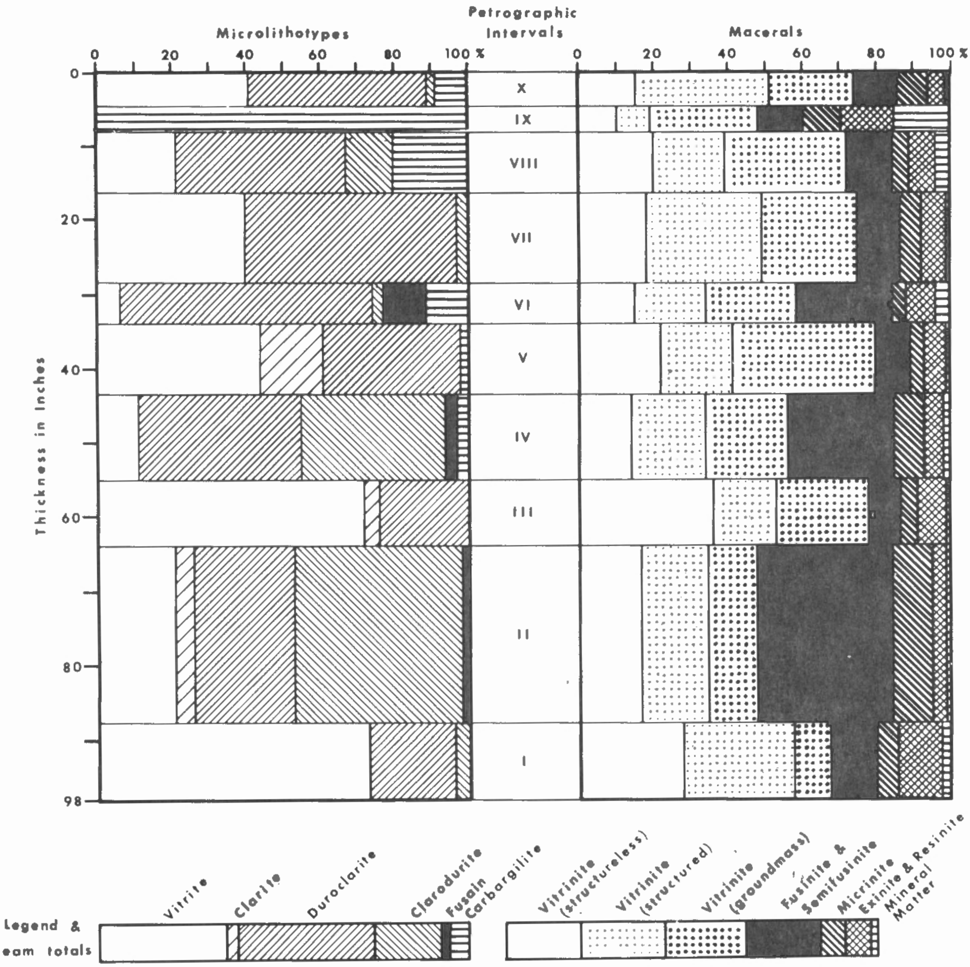

Figure 2. Petrographic composition of lignite column CQ45, Estevan, Saskatchewan.

III while the former is in interval IX which is high in carbargilite. The average content of bright microlithotypes for the seam is 37 per cent. The dullest intervals are II, IV, VI and IX. The over all aspect of sample CQ 45 is that of an intermediate to dull coal.

In terms of macerals vitrinite is the most abundant with an average of 64 per cent for the whole seam, almost equally divided among the three categories. The range of variation between intervals is 48 to 80 per cent with intervals II and V being lowest and highest respectively. Structured vitrinite is most abundant in intervals I and X at the top and bottom of the seam and in interval VII. It often contains resinite or resinite-like bodies (Pl. I, figs. 1, 2). Sometimes it is difficult to distinguish from semi-fusinite; in other instances it seems to grade into structureless vitrinite (Pl. I, fig. 4). Structureless vitrinite is widely distributed in all intervals. Groundmass vitrinite appears to be more abundant in the upper half of the seam. As shown in Plate II, figs. 1, 2 and 3, it typically has a lower reflectance than the other two vitrinite varieties, a mottled or granular appearance and is the matrix for a variety of nonvitrinite components. Fusinite and semifusinite are abundant constituents in this coal and the over all seam content is 19 per cent. These constituents are especially concentrated in the lower 2/3's of the seam and particularly in intervals II, IV and VI. These units alternate with brighter intervals containing less opaque constituents and more vitrinite. Although the contents of the macerals fusinite and semi-fusinite are high, that of the microlithotype fusite is relatively low. This suggests that the fusinite and semifusinite are widely dispersed in small masses in those intervals in which their contents are high rather than being concentrated in distinct relatively thick layers. Resinite of various kinds is a common constituent and is often associated with the structured vitrinite (Pl. I). Resinite grains often occur also as isolated bodies or as clusters in association with the groundmass vitrinite (Pl. III, fig. 1). Exinite, that is the coalified remains of spores, pollen and cuticles, is a fairly common constituent in this coal, reaching a high of 16 per cent in interval IX. It most commonly is associated with the groundmass vitrinite (Pl. II, figs. 1, 2).

The chemical data are given in Table 1 and their variations in volatile matter, ash, ultimate carbon, hydrogen and sulphur through the height of the seams are plotted in Figure 3. The ash data show that this seam is composed of relatively clean coal averaging 6-8 per cent ash on the dry basis in the lower seven intervals. Interval IX which is made up largely of carbargilite and impure coal has nearly 20 per cent ash while the uppermost interval X has approximately 10 per cent. The sulphur content is low and shows a high of 0.88 per cent on the dry, ash-free basis in interval III. The profile for sulphur in Figure 3 shows that the lower five intervals are slightly higher on the average than are the upper five units. The profiles for hydrogen and volatile matter show several corresponding peaks and depressions which appear to be related to petrographic composition. The three intervals high in fusinite (II, IV and VI - especially the first two) show depressions. Interval IX in comparison shows the highest exinite content and also the highest volatile matter and hydrogen contents. Attempts were made to explore further the relationship between petrographic and chemical compositions but many of these relationships are not readily apparent. For example, the literature contains many references to the hydrogen-rich character of exinite and resinite. Plots of the data in the present study failed however to show more than general trends in this direction. Plots of volatile matter

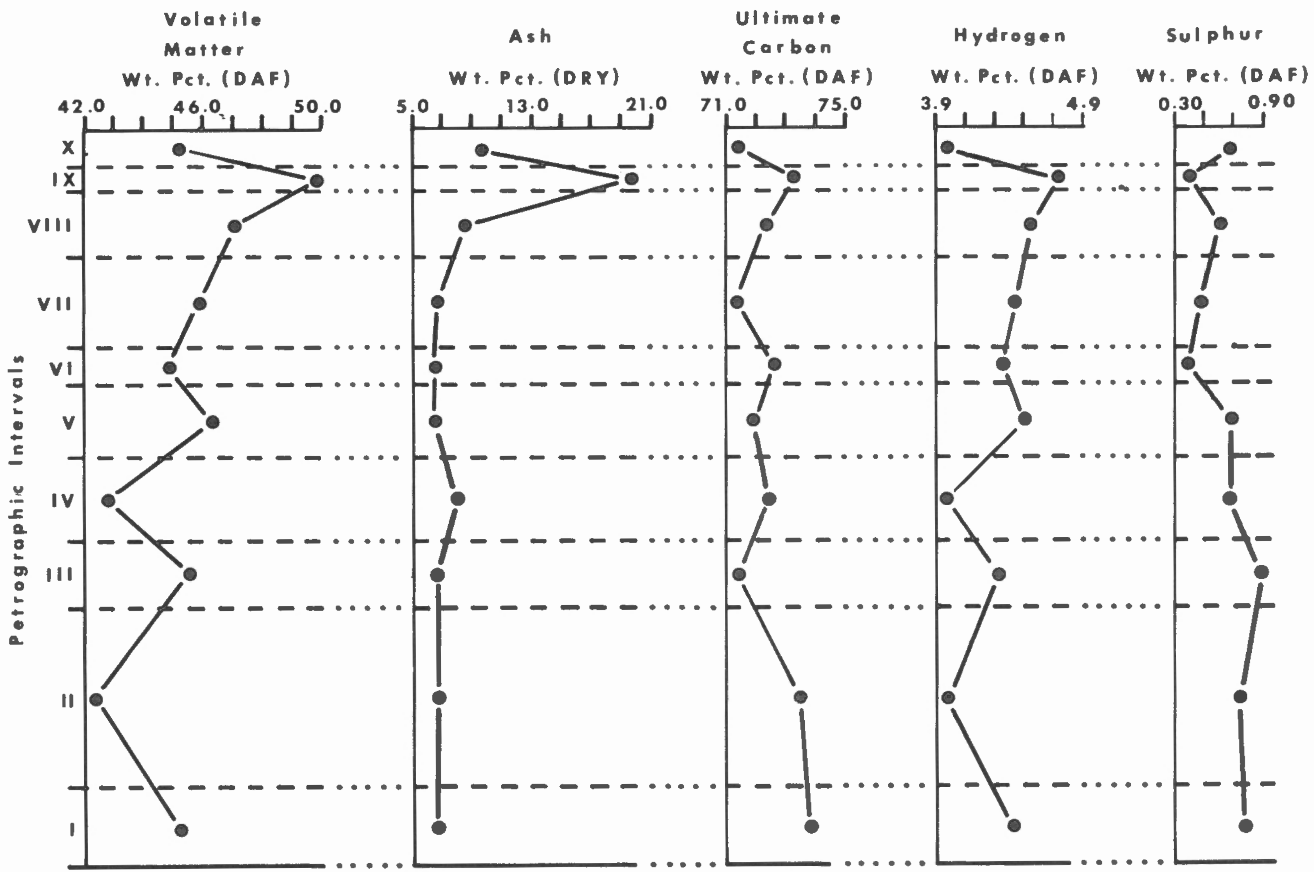

Figure 3. Chemical composition of intervals in CQ 45.

Table 1. Proximate and Ultimate Analyses for Intervals of CQ45 (Estevan)

Interval	Moisture and ash basis	Moisture	Ash	Volatile Matter	Fixed Carbon	Ultimate Carbon	Hydrogen	Sulphur	Nitrogen	Oxygen
	As Rec'd	11.44*	8.66	35.99	43.91	57.09	3.18	0.54	0.90	18.19
X	Dry	-	9.78	40.64	49.58	64.46	3.59	0.61	1.02	20.54
	DAF	-	-	45.04	54.96	71.44	3.98	0.68	1.13	22.77
	As Rec'd	9.67	18.00	36.05	36.28	52.97	3.41	0.29	0.86	14.80
IX	Dry	-	19.93	39.91	40.16	58.64	3.78	0.32	0.95	16.38
	DAF	-	-	49.84	50.16	73.24	4.71	0.40	1.19	20.46
	As Rec'd	12.91	7.50	37.56	42.03	57.61	3.60	0.48	0.95	16.95
VIII	Dry	-	8.61	43.13	48.26	66.15	4.13	0.55	1.09	19.47
	DAF	-	-	47.19	52.81	72.38	4.52	0.60	1.19	21.30
	As Rec'd	13.05	6.08	37.13	43.74	57.59	3.57	0.39	0.94	18.38
VII	Dry	-	6.99	42.70	50.31	66.23	4.11	0.45	1.08	21.14
	DAF	-	-	45.91	54.09	71.21	4.42	0.48	1.16	22.73
	As Rec'd	12.61	5.81	36.54	45.04	59.16	3.55	0.32	0.84	17.71
VI	Dry	-	6.65	41.81	51.54	67.70	4.06	0.37	0.96	20.26
	DAF	-	-	44.79	55.21	72.52	4.35	0.39	1.03	21.71
	As Rec'd	13.10	5.72	37.62	43.56	58.41	3.65	0.56	0.84	17.72
V	Dry	-	6.58	43.29	50.13	67.22	4.20	0.64	0.97	20.39
	DAF	-	-	46.34	53.66	71.95	4.50	0.69	1.03	21.83
	As Rec'd	13.90	6.89	33.72	45.49	57.39	3.14	0.54	0.85	17.29
IV	Dry	-	8.00	39.16	52.84	66.66	3.65	0.63	0.99	20.07
	DAF	-	-	42.57	57.43	72.45	3.96	0.68	1.07	21.84
	As Rec'd	14.54	5.80	36.26	43.40	56.57	3.43	0.70	0.92	18.04
III	Dry	-	6.79	42.43	50.78	66.19	4.01	0.82	1.08	21.11
	DAF	-	-	45.52	54.48	71.01	4.31	0.88	1.16	22.64
	As Rec'd	13.27	5.40	34.07	46.76	59.37	3.21	0.57	0.94	16.74
II	Dry	-	6.80	39.28	53.92	68.45	3.70	0.66	1.08	19.31
	DAF	-	-	42.15	57.85	73.45	3.97	0.71	1.16	20.71
	As Rec'd	13.41	5.94	36.44	44.21	59.44	3.56	0.62	0.74	16.29
I	Dry	-	6.86	42.08	51.06	68.65	4.11	0.72	0.85	18.81
	DAF	-	-	45.18	54.82	73.70	4.41	0.77	0.92	20.20

* Samples submitted for proximate analysis were air dried.

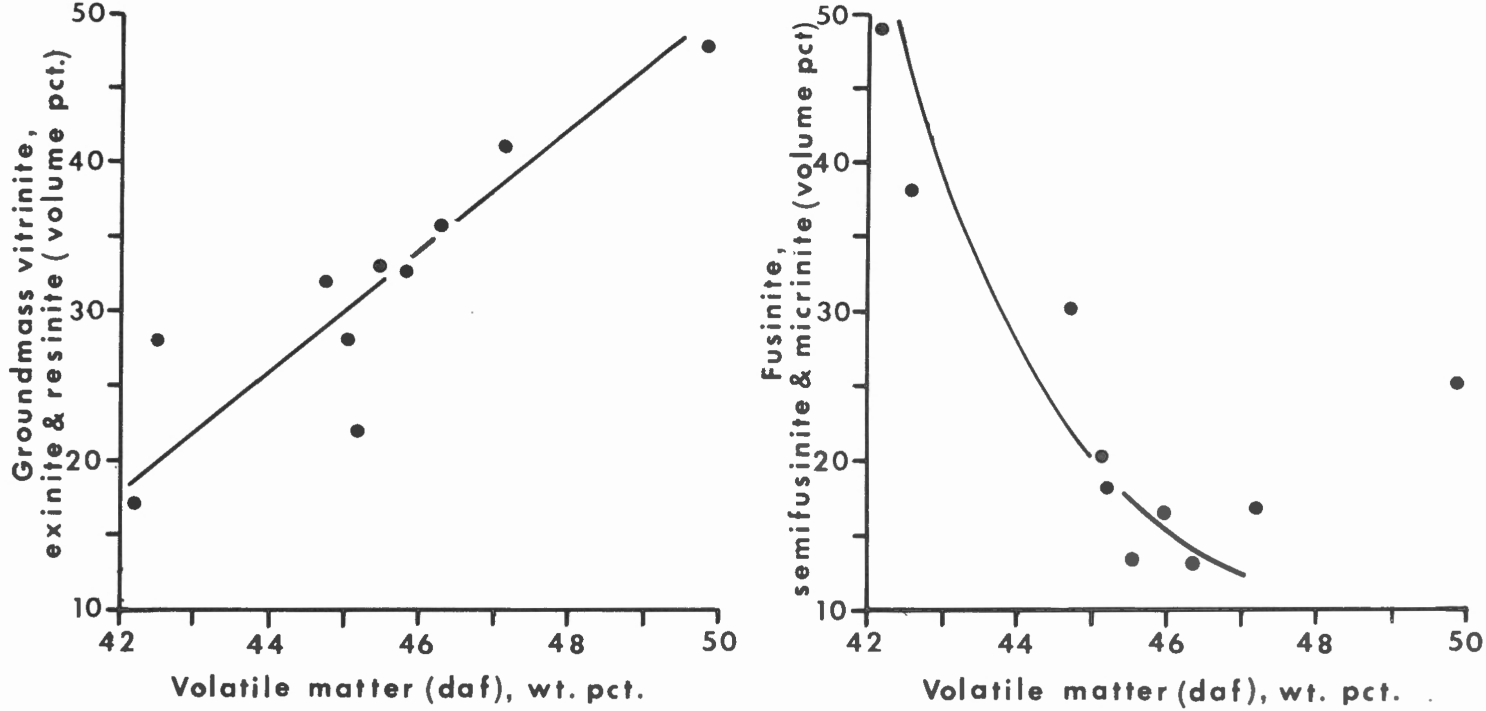

Figure 4. Relation of volatile matter to petrographic composition.

against exinite and resinite plus groundmass vitrinite (Fig. 4) showed a somewhat better correlation while a plot of volatile matter against the so-called inert or carbon-rich constituents (fusinite, semifusinite and micrinite) showed the reverse trend of higher volatile matter with lower inert content (Fig. 4).

Teichmüller and Thomson (1958) studied and compared the petrographic, palynological and chemical compositions of important facies types in the Rhine brown coal. They recognized three main facies types of coal, derived from three different depositional environments, namely forest-moor, reed-moor and open-water. They showed that the forest-moor coals contained relatively high proportions of both resin-saturated and nonresinous cellular tissue along with smaller amounts of gel and fusinite. The total of these constituents in the coals that they assigned to the forest-moor facies approached 50 per cent while the remainder was constituted of "humic detritus" or strongly decomposed remains of tissues, especially wood, forming a groundmass. The reed-moor coals differed in that the proportions of humic detritus greatly increased to 80 per cent or more. They found that higher concentrations of fusinite generally occur in the forest-moor coals

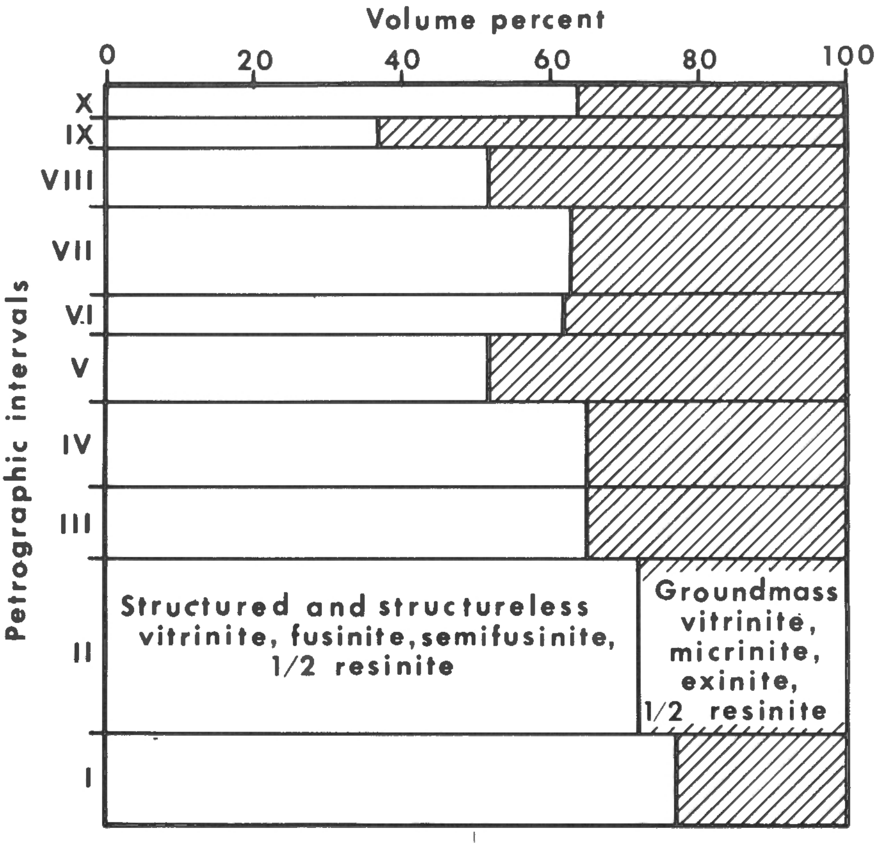

Figure 5. Distribution of petrographic constituents in CQ 45 for correlation with environments of deposition.

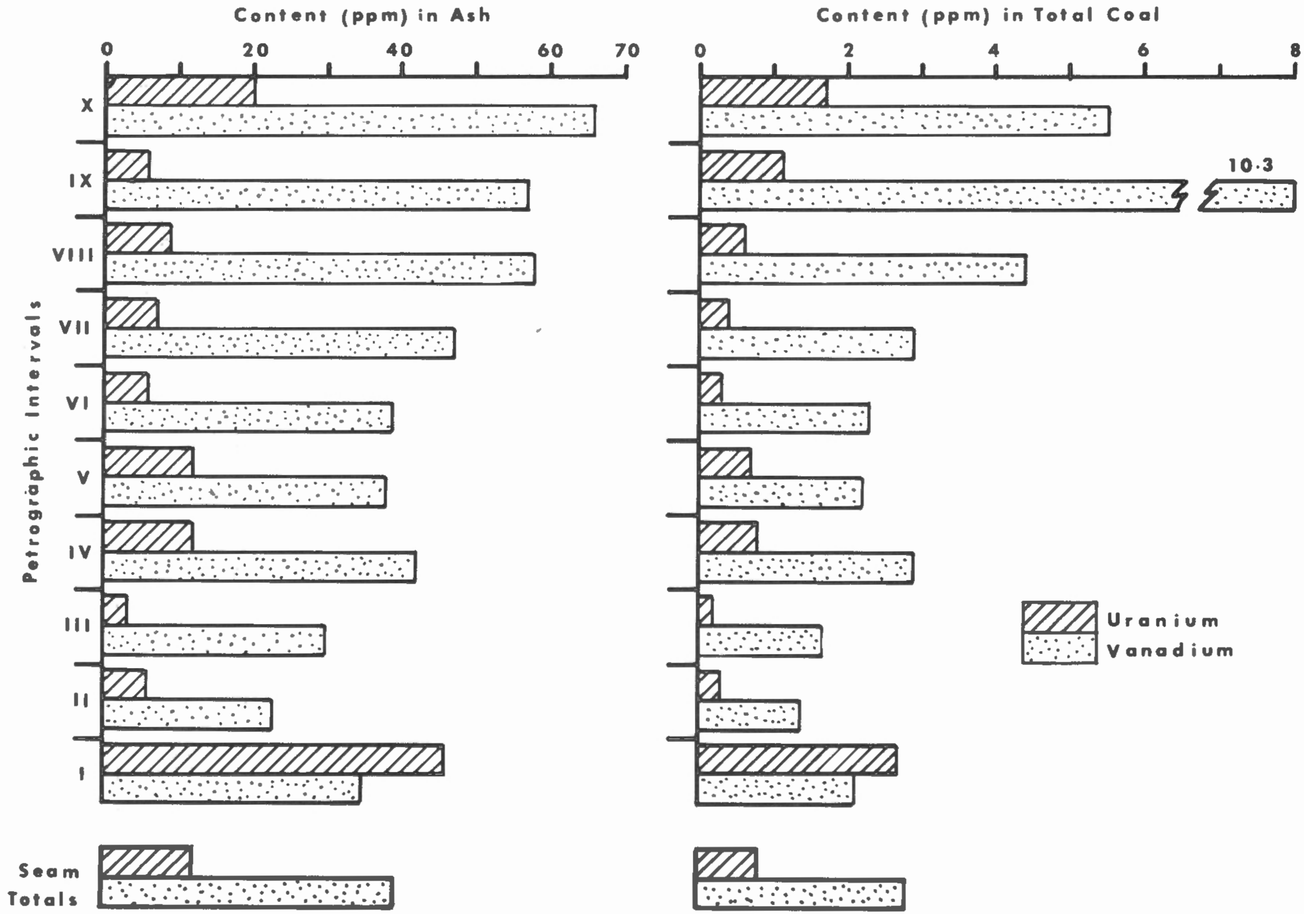

Figure 6. Distribution of uranium and vanadium in intervals of sample CQ 45.

wheras exinite tended to be increased in the reed-moor coals. Open-water coals or "gyttja"-type deposits tended also to be high in humic detritus and exinite along with high ash. Figure 5 shows a plot of the maceralic data of the intervals of CQ 45 with these data regrouped in line with the ideas of Teichmüller and Thomson. Structured vitrinite, "gel" or structureless vitrinite, fusinite and semifusinite have been grouped together as one entity and the groundmass vitrinite (humic detritus) along with exinite, resinite and micrinite have been grouped as a second major entity. Although this must represent an approximation only because the present study was not done in the detail of the Rhine coal study and the ranks of coal involved are different, is is nevertheless interesting to compare the results with Teichmüller and Thomson's data. Going from their data to that on the Estevan column, it would seem that the higher the content of structured and structureless vitrinite, fusinite and semifusinite, the greater the possibility of a forest-moor source for the coal in question. Looked at in this light the petrography suggests strongly that the Estevan lignite is the product of a forest-moor environment with such influences being strongest toward the base of the seam especially intervals I and II. The abundance of fusinite and semifusinite in some intervals (II, IV and VI) might be indicative of some type of subdivision within the forest-moor environment as Teichmüller and Thomson suggest. Interval IX on the other hand has most of the characteristics of an open-water deposit according to Teichmüller and Thomson's description. This interval has a high exinite content, a fairly high content of groundmass vitrinite, high ash and one of the highest nitrogen contents in the seam (see Table 1).

ANALYSIS FOR URANIUM, VANADIUM AND MOLYBDENUM

The ash residues from the proximate analysis of the interval samples were subjected to spectrochemical analysis for uranium, vanadium and molybdenum. For the first two elements standard emission spectrographic techniques were followed modified somewhat because of the small amount of ash in some of the intervals. Uranium contents were determined fluorimetrically. In Figure 6 the uranium and vanadium contents of the intervals have been plotted and are represented by both the ppm in the ash and the ppm on the total coal basis. Molybdenum values for all ten samples was reported as less that 100 ppm on the ash. Because of the limitations of the method for this particular element accurate quantitative determinations cannot be carried out for molybdenum concentrations of less than 100 ppm.

One hundred parts per million is at the lower limit of what Krauskopf (1955) considered an average content of molybdenum for coal ash. Vanadium values were also low and ranged from 23 to 66 ppm on the ash. This is also lower than the average content for coal ash according to Krauskopf. Uranium values varied from 3 to 46 ppm on the ash or 0.2 to 2.7 ppm on the total coal basis. According to Vine (1962) the average abundance of uranium in coaly carbonaceous rocks is normally less than 0.0001 per cent (1 ppm).

It can be seen that the higher uranium values tend to occur at the bottom and top of this seam (Intervals I and X respectively). This concentration at the margins suggest sorption by the organic material from circulating groundwater after seam burial according to Nicholls (1968). The low values for uranium check with the generally low values for this element reported by Cameron and Birmingham (1970) for the Estevan area.

Vanadium contents are also unequally distributed among the intervals of CQ 45 and tend to concentrate at the top of the seam. Zubovic et al. (1961)

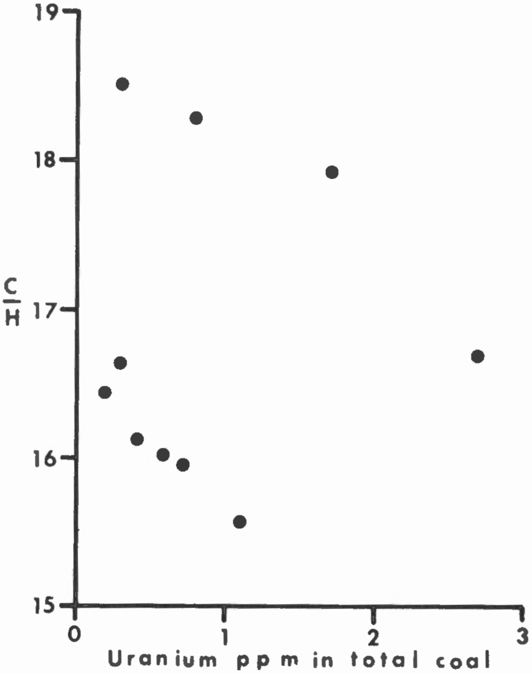

Figure 7.
Relation of uranium content to carbon-hydrogen ratio.

reporting on minor element contents in a column sample of lignite from North Dakota collected 15-20 miles south of Estevan, also showed low values with nearly all their intervals within the seam averaging 8 ppm or less on the total coal basis. However an exception in their column was a basal unit with high ash which gave 31 ppm vanadium on the total coal basis. According to Nicholls (1968), vanadium is most often associated with the inorganic fraction of the coal, that is its content on a total coal basis rises with increasing ash content. There is some suggestion that this holds for CQ 45 because interval IX with 18.00 per cent ash has the highest content of 10.3 ppm vanadium on the total coal basis.

Attempts to relate vanadium content to petrographic parameters were inconclusive. Attempts to correlate uranium content with the contents of various macerals and combinations of macerals and chemical values showed no clear cut relationship with one exception. When the carbon/hydrogen ratios of the intervals were plotted with uranium content a curious relationship appeared as shown in Figure 7. In this instance the points representing different intervals sorted themselves into two rather distinct populations both of which show an increase of uranium content as the C/H ratio decreases. The only petrographic or chemical property (other than C/H ratio) which seems to distinguish the upper four intervals from the lower six as plotted in Figure 7 is that the upper four all contain large proportions of the structured constituents that is, structured vitrinite, fusinite and semifusinite (43-55%) in comparison with the lower six (28-43%). Because a large proportion of the structured material consists of high-carbon fusinite and semifusinite this could explain the higher C/H of the upper four intervals but the rather neat development of uranium distribution patterns with both upper and lower populations must be related to some other as yet unexplained factor.

PLATES

PLATE I

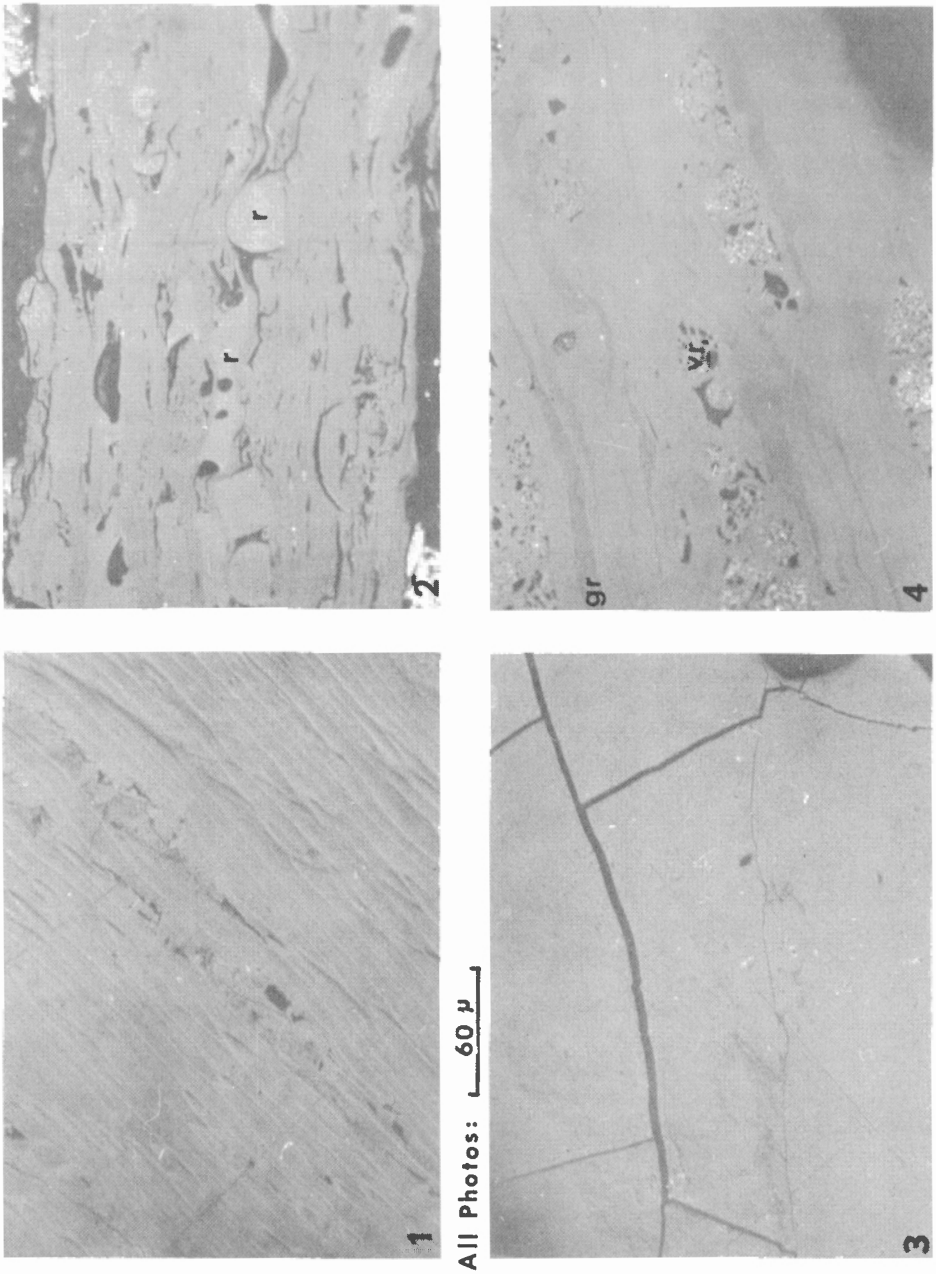

PLATE I

1. Structured vitrinite.

2. Structured vitrinite with resinitic inclusions(r).

3. Structureless vitrinite; note the relatively uniform texture, the cracks and the relatively high reflectance.

4. Structured vitrinite which appears to be transitional to structureless vitrinite; the photo shows resinitic inclusions, some with vacuoles (vr), some lens-like and of a darker grey colour (gr).

All photos; reflected light, oil immersion.

PLATE II

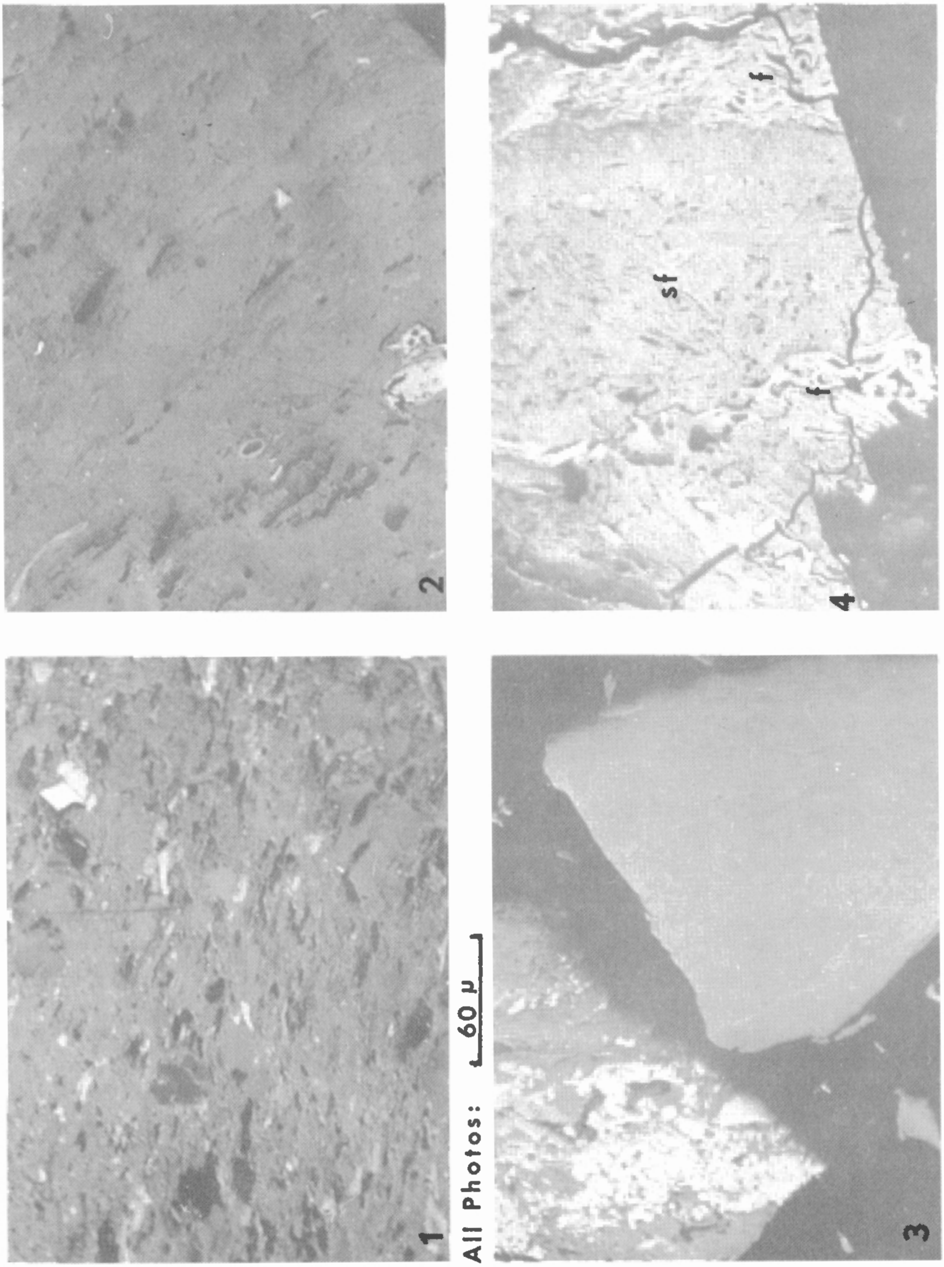

PLATE II

1. Groundmass vitrinite forming a matrix for a variety of other constituents namely, micrinite (small white masses), exinite (black elongate bodies), resinite (grey to black ovoid and irregularly shaped bodies).

2. Groundmass vitrinite with exinite (small black to dark grey elongate bodies).

3. Large fragment of structureless vitrinite in lower right hand side of photo. Second large fragment consists of groundmass vitrinite with a high content of micrinite (white masses). Note higher reflectance of the structureless vitrinite when compared with the groundmass vitrinite.

4. Fusinite (f) and semifusinite (sf).

All photos: reflected light, oil immersion.

PLATE III

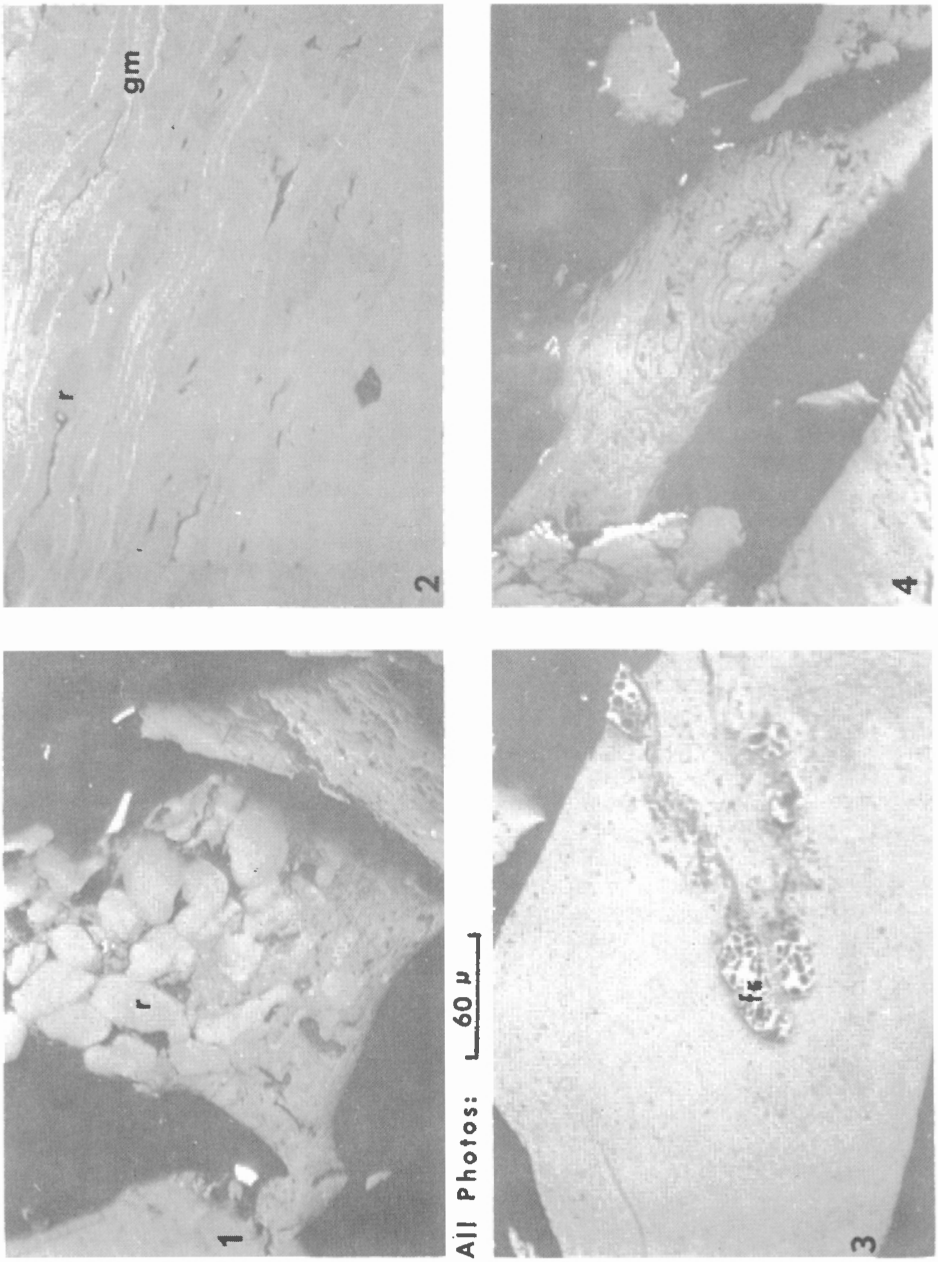

PLATE III

1. Cluster of resinite-like bodies (r) associated with groundmass vitrinite.
2. Stringers of granular micrinite (gm) associated with structureless vitrinite and resinite (r).
3. Fungal remains? (fr) or carbonized resin associated with structureless vitrinite.
4. Fragment in centre contains possible remains of cork tissue. Such material in the present study was classified as structured vitrinite.

All photos: reflected light, oil immersion.

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